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ABSTRACT

The Zone Melt Crystal Growth Method (FSZM) has been used to produce polycrystalline Galfenol specimens, $Fe_{81.6}Ga_{18.4}$, with preferred {100} orientation. This crystal growth technique has advantages over conventional Bridgman methods in that zone rates used were at least an order of magnitude greater; 350 mm/hr versus 2-4 mm/hr. This material had measured magnetostrictions ranging from 168 ppm to 220 ppm compared to 290 ppm for a single crystal with a similar composition. It was discovered that upon machining a large increase in magnetostriction occurred, ~15%. Using Orientation Imaging Microscopy (OIM) techniques it was shown that the magnetostriction increase is due to the removal of off-axis grains located on the circumference of the FSZM samples. The room temperature mechanical properties were measured to be 72.4 GPa – 86.3 GPa modulus of elasticity, 348 MPa – 370 MPa ultimate strength, and elongation values of 0.81% - 1.2% depending upon zoning conditions.

Keywords: Galfenol, iron-gallium alloys, magnetostriction, structural materials, transduction, OIM

1. INTRODUCTION

It has been shown that a partial substitution of Fe atoms with Ga atoms, in the Fe bcc crystal structure, creates an active material with approximately ten times the magnetostriction of pure Fe. This material has been named Galfenol for the Gallium addition to the iron (Fe) matrix discovered by the Naval Surface Warfare Center - Carderock Division (NSWC-CD) formerly known as the Naval Ordinance Labs (NOL).[1]

Galfenol has been grown using conventional Bridgman growth processing techniques to produce single crystals of various compositions.[1-6] The production of single crystal samples has allowed for detailed analysis of the various crystallographic directions present in the body centered cubic structure. From past research of iron single crystals it has been determined that the <100>, <110>, and <111> family of directions are the magnetically “easy”, “medium”, and “hard” directions. The study of single crystals has allowed the ideal or maximum magnetostrictions to be determined for this material with various gallium contents.

Magnetostriction values of Galfenol have been reported over a wide composition range, $Fe_{1-x}Ga_x$ ($0.04 \le x \le 0.35$). Only Galfenol material with Ga values less than 22 at% will be discussed in this paper due to the fact that the maximum in magnetostriction[6] occurs at approximately 19 at% Ga and alloys with higher gallium contents have not been fully explored. Clark et al.[1] determined the magnetostriction values for [100] single crystal $Fe_{83}Ga_{17}$, at 50 MPa (~7 ksi) pre-stress, to be ~275 ppm. Kellogg et al.[4] reported the magnetostriction for a [100] single crystal $Fe_{81}Fe_{19}$ to be ~320 ppm at 45 MPa (~6.5 ksi) pre-stress. It should be noted that in addition to a post-growth annealing step Kellogg also quenched the crystal to obtain a random distribution of Ga atoms on the Fe lattice. In another publication Clark et al.[5] reported similar magnetostriction values for a [100] single crystal $Fe_{81}Ga_{19}$ at 50 MPa (~7 ksi) pre-stress. For a [100] $Fe_{79}Fe_{21}$ single crystal the magnetostriction dropped to ~200 ppm, at 41 MPa (~6 ksi) pre-stress with the decrease in magnetostriction with increasing Ga content being attributed to the partial formation of the ordered DO_3 phase displacing the disordered α-Fe structure responsible for the large magnetostriction increases. Once again a post-growth quench treatment was performed on this sample however it was not effective in preventing long-range ordering of the Ga. For comparison purposes the magnetostriction of Fe and Terfenol-D has been reported to be 20 ppm and over 1000 ppm, respectively.[7]

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The relatively low magnetostriction values for Galfenol when compared to giant magnetostrictives such as Terfenol-D would beg the question "Why use Galfenol?" The answer to this question comes when we begin to examine and understand Galfenol's mechanical properties. Terfenol-D, like other popular active materials (i.e. piezo ceramics) are quite brittle in nature; while they operate well in compression their tensile strength values are less than 15ksi for piezo materials and 4 ksi for Terfenol-D. They could not be used in tension, transduction, or actuation situations in-which the environment is prone to large shocks or other violent disturbances.

Polycrystalline Galfenol materials, of a $Fe_{83}Ga_{17}$ composition, has been shown to exhibit tensile strengths up to 440 MPa (~64 ksi) with elongations of 0.25% before failure.[8] Clark et al.[1] reported the stiff Young's modulus (low field, high stress) for a [100] single crystal of $Fe_{83}Ga_{17}$ to be ~77 GPa (11.1 Msi). Vickers hardness values also were reported to be between 200 and 250 for Ga content between 15 at% and 20 at%. At higher Ga content, in excess of 30 at% the Vickers hardness values exceed 475. These hardness values imply that the material is rugged in nature with good ductility. In Kellogg's publication[9] he reported on the static mechanical property values for $Fe_{83}Ga_{17}$ single crystals. A [100] oriented single crystal had a reported modulus of 65 GPa (9.4 Msi), a tensile strength of 515 MPa (74.6 ksi), and an elongation value >2%, however, fracture occurred shortly after this value of strain was reached. Compare these mechanical property values with Terfenol-D which has a Young's Modulus of 90 GPa (13 Msi) and a tensile strength of only 28 MPa (4 ksi), but has a high magnetostriction or compare the Galfenol values to iron which has elongations greater than 20%, but poor magnetostriction. Galfenol offers better magnetostriction than the ductile active materials such as iron and improved mechanical properties when compared with giant magnetostrictives such as Terfenol-D.

As stated earlier Galfenol materials have been produced using slow growth methods in a conventional Bridgman process. This paper examines Galfenol material produced using the Zone Melt Crystal Growth Method (FSZM). The FSZM process allows the production of a large number of grain-oriented (or growth-textured) Galfenol samples at faster growth rates compared to the Bridgman process. Galfenol samples discussed in this paper are described as either Research grade or Production grade Galfenol. The only difference between the two types of materials is the rate at which they were zoned. Research grade material is zoned at slower rates than production grade material, but these rates are at least an order of magnitude faster than used to produce single crystal Galfenol via the Bridgman process.[1-6, 9]

2. SAMPLE PREPARATION AND MAGNETIC MEASUREMENTS

Necessary amounts of electrolytic iron (99.99% pure) and gallium (> 99.99% pure) were measured out to produce Galfenol with approximately 18.4 at% (22 wt %) gallium composition. This composition was chosen to simplify the production process by reducing the risk of DO_3 phase formation during solidification. By choosing a lower gallium content alloy thermal treatments could be avoided. The metastable phase diagram produced by Ikeda et al.[10] was used as a reference for choosing this composition.

The raw materials were arc melted several times under a slight negative pressure to homogenize the material. The resulting 170 gram buttons were then arc melted into a feedstock mold to form rough feedstock with a 10 mm+ diameter and 260 mm length. The rough feedstock was then ground on a belt grinder to produce the final feedstock form of 9.5 mm diameter and 255 mm length and prepped for FSZM.

Research grade, $Fe_{81.6}Ga_{18.4}$, Galfenol specimens were FSZM under inert atmosphere at elevated pressures to minimize gallium loss. Zone rates in excess of 25 mm/hr were used to produce the Research grade material. Production grade Galfenol specimens were zoned under the exact same conditions except zone rates used were in excess of 350 mm/hr. Compare these zone rates to the 2 mm/hr – 4 mm/hr Bridgman rates used to produce single crystal material above. The FSZM Galfenol rods were then sectioned into standard size test specimens with dimensions of 9.35 mm diameter and 50 mm long. Some samples had the ends ground and polished for microstructure analysis and grain size determination.

The strain performance/magnetostriction was measured for all samples. Magnetostriction measurements were taken on a conventional dead-weight test stand where the mechanical preload is applied axially and can vary from 6.9 MPa to 69 MPa. The samples were placed inside a coil of an electro-magnet. A current is applied to the coil which induces a magnetic field causing the Galfenol to magnetostrict. The current is produced by a power amplifier/signal generator, at very low frequency of 0.1 Hz to minimize the inductive effects of the coil. During the testing of the Galfenol samples, the peak current was as high as 4 amps and the magnetic fields produced were up to 1.5 kOe. Magnetic field intensity (H-field) was measured by placing a Hall-effect generator near the center of the sample. The magnetic flux density (B-field) is measured simultaneously, which allows the magnetic permeability to be measured. This is done by winding the pick-up coil of a magnetic fluxmeter directly on the sample. A linear variable displacement transducer (LVDT) is used to sense the change in length or magnetostriction of the sample.

Upon completion of testing the Galfenol samples were center less ground to a final diameter of 6.35 mm and the ends ground parallel to produce a final length of ~50 mm. Final magnetostriction testing is done after this machining step. The reason for the second round of testing will be discussed in detail in the Results and Discussion section.

3. RESULTS AND DISCUSSION

Chemical analyses for arc melted and FSZM material were measured by Inductively Coupled Plasma – Mass Spectrometry (ICP-MS) by Northern Analytical Laboratories. Weight losses during processing were found to be minimal with the final gallium contents, after FSZM, reported between 18.1 at% Ga and 18.4 at% Ga.

Microstructures of the research grade and production grade Galfenol were examined to determine grain size and shape. Samples, with the zoning direction normal to the sample, were prepared by standard metallographic methods. A Nital etchant was used to prepare the surface for examination. The average grain size for the research grade samples was measured to be 1350 µm ± 150 µm; and it should be noted that several of the samples contained large single grains several thousand microns in diameter. The average grain size for the production grade samples was measured to be 630 µm ± 85 µm. As expected, faster rates resulted in a smaller grain size due to increased solidification rates. Figures 1 and 2 below are example micrographs of a research grade and production grade sample, respectively. Both seem to exhibit a standard shape grain microstructure. In Figure 2 sub-grain boundaries within each grain are clearly evident. Sub-boundaries usually exhibit crystal lattice mis-orientations of approximately 1º and are not believed to affect the materials properties.

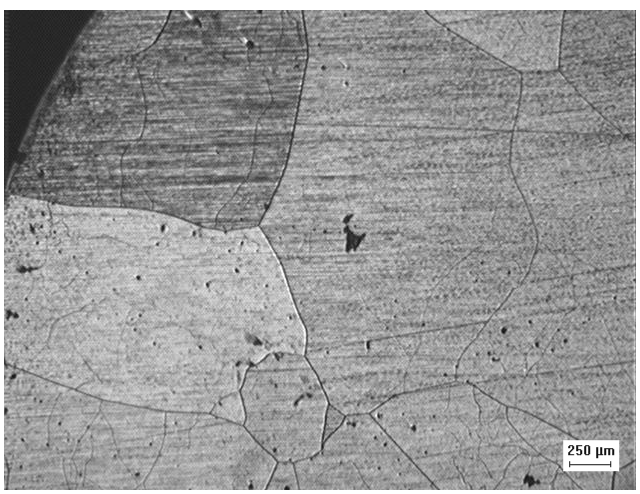

Fig. 1. Research grade sample microstructure.

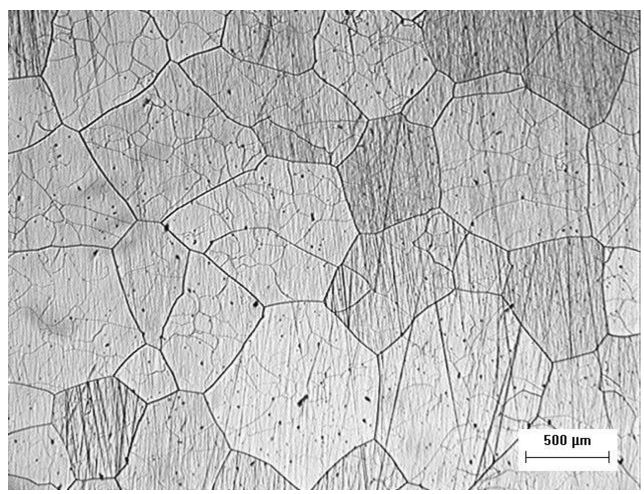

Fig.2. Production grade sample microstructure.

The 18.4 at% Ga composition was chosen to minimize the risk of ordered DO_3 phase formation. Powder x-ray diffraction measurements were performed to verify that the formation of the ordered DO_3 phase was not present in either research or production grade material samples. Due to the similarity of atomic scattering factors of Fe and Ga, intensities of the superlattice reflections arising from DO_3 ordering are extremely weak (<1% max), requiring more precise measurements to resolve the superlattice reflections from background levels. Figure 3 shows a typical x-ray scan for a production Galfenol specimen; inset within the scan is a close-up of the location of the two superlattice reflections arising from DO_3 ordering. As can be seen, the DO_3 phase is not present in the production grade sample. Similar results have been attained for the research grade material despite the slower growth rates.

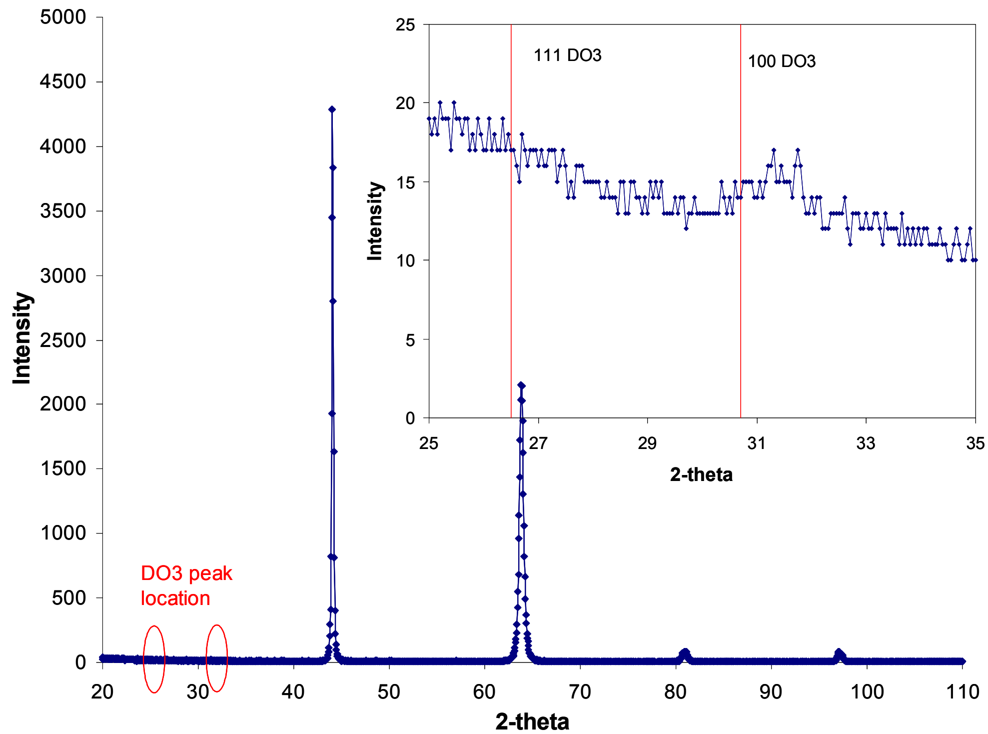

Fig. 3. Standard x-ray scan of a production grade Galfenol sample showing absence of DO_3 phase.

3.1 Magnetostriction and machining effects

Bulk magnetostriction results were measured for several research grade and production grade samples in the as-zoned condition. Magnetostriction values were recorded at ~50 MPa (7 ksi) pre-stress in a 500 Oe magnetic field; which is beyond the magnetic saturation point for this material. As-zoned research grade material had measured magnetostrictions of 190 ppm ± 25 ppm, while production grade material had measured magnetostrictions of 149 ppm ± 12 ppm. Single crystal values for similar compositions range from 275 ppm to 320 ppm; however, they were produced by a slow growth Bridgman process and the high composition samples required quench treatments to achieve these high magnetostrictions.

In order to obtain accurate magnetization data rods were machined to ensure constant diameter and parallelism. After machining significant improvements in magnetostriction were found for all samples. Research grade material had an average of 15% increase in magnetostriction (220 ppm ± 25 ppm) with a maximum increase of 39%, while the production grade material also had an average increase of 15% (168 ppm ± 18 ppm) with a maximum increase of 24%. Figures 4 and 5 show typical magnetostriction plots for research grade and production grade Galfenol before and after machining. The magnetization values for both research grade and production grade was measured at ~1.5 tesla. This result is well within the published magnetization range of 1.3 tesla to 1.8 tesla for Galfenol single crystal specimens of various compositions.

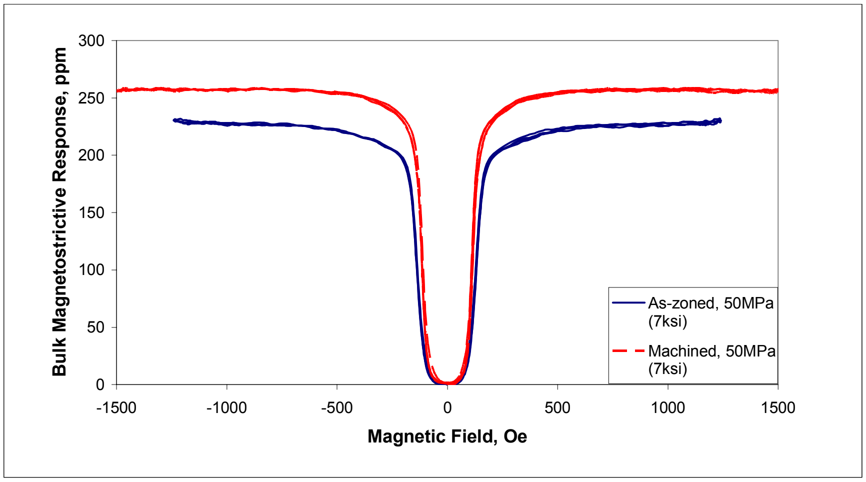

Fig. 4. Research grade Galfenol bulk magnetostrictives response, before and after machining.

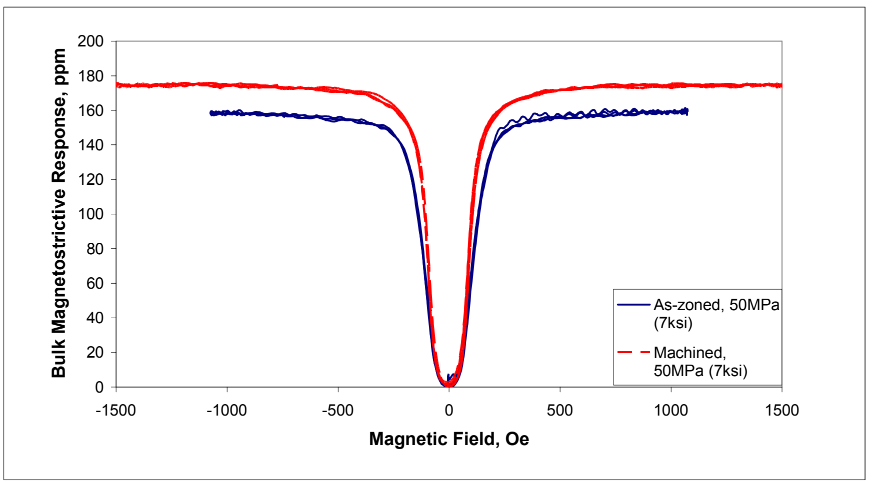

Fig. 5. Production grade Galfenol bulk magnetostrictives response, before and after machining.

It was hypothesized that during FSZM, grains in the outer area of the samples oriented themselves in non-ideal crystallographic directions (i.e. not <100>) due to faster solidification rates. The misalignment between the {100} oriented grains, in the interior of the samples, and the non-ideal grains would lead to the creation of internal stresses during magnetization thus lowering the bulk magnetostriction of the material. During machining the majority of these mis-oriented grains were removed resulting in an improvement in the bulk magnetostriction. In order to evaluate this hypothesis two research grade samples, from the same FSZM rod, one as-zoned and one machined were analyzed using Orientation Imaging Microscopy (OIM) techniques. The Electron Back-Scattered Diffraction (EBSD) analysis was performed by HKL Technology using their Nordlys EBSD detector. A similar analysis was performed on two production grade samples from the same FSZM rod.

Figures 6 and 7 are orientation maps of an as-zoned and machined research grade Galfenol samples. The zoning direction normal to the analyzed surface. The as-zoned sample, Figure 6, has a dashed circle drawn on it to indicate where the machined diameter would be located. Both figures show strong {100} orientation with the as-zoned sample having a measured magnetostriction of 190 ppm and the machined sample having a measured magnetostriction of 255 ppm. However, in Figure 6 one large off-axis grain can be identified on the outer diameter along with a handful of smaller off-axis grains. The orientation map of the machined sample, Figure 7, is almost 100% oriented along the

<100> direction. It should also be noted that the measured misorientations for the {100} grains in the machined sample were less than 5 degrees; the {100} grains in the as-zoned sample had misorientations of up to 10 degrees, validating the hypothesis that the grains with the largest misorientations are located around the circumference of the as-zoned sample.

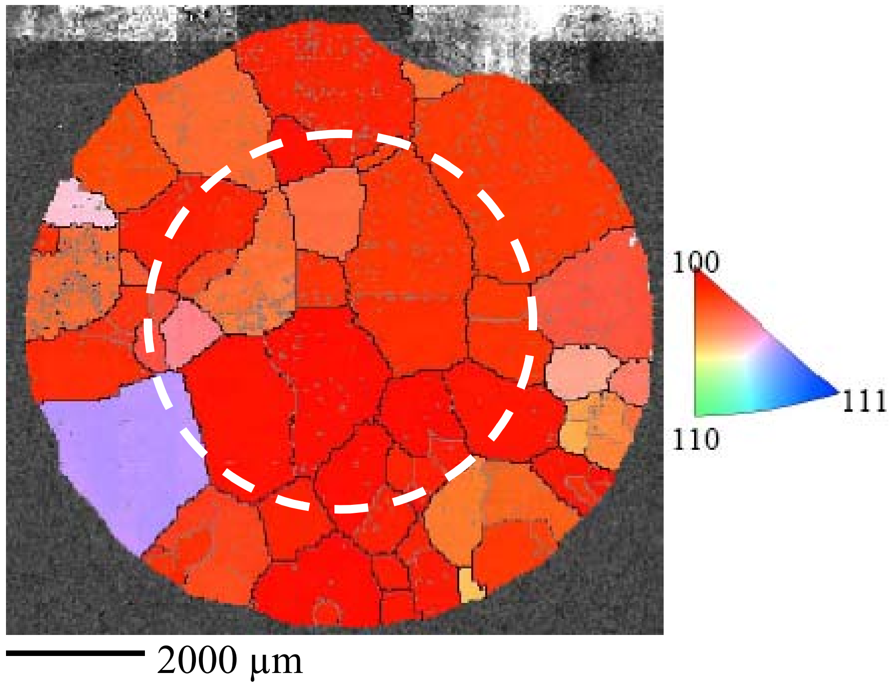

Fig. 6. Orientation map of as-zoned research grade sample. Note the large off-axis grain on outer diameter.

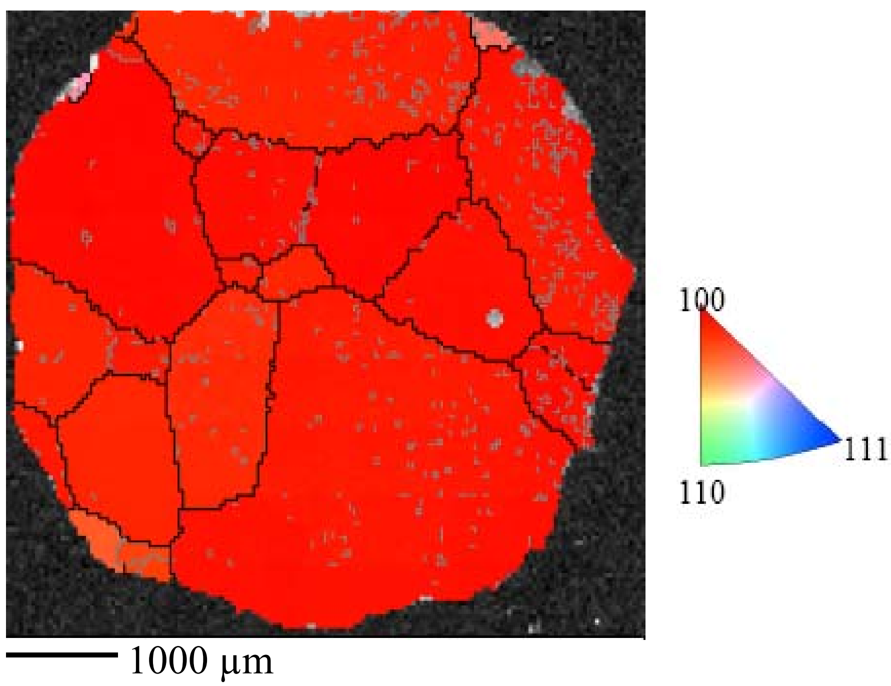

Fig. 7. Orientation map of machined research grade sample. Notice that 95+% of sample is oriented along {100}.

The pole figures for both samples are shown in Figures 8 and 9. Both have high maximum relative intensities greater than 11, which is indicative of strong fiber textrue, in this case {100}. The machined sample has a maximum relative intensity of 20.68 while the as-zoned sample has a maximum relative intensity of 11.51.

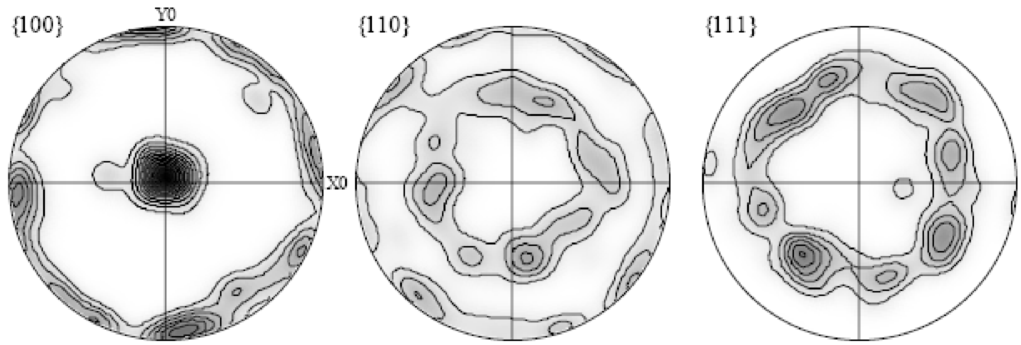

Fig. 8. Pole figures for as-zoned research grade sample. Relative maximum intensity of 11.51.

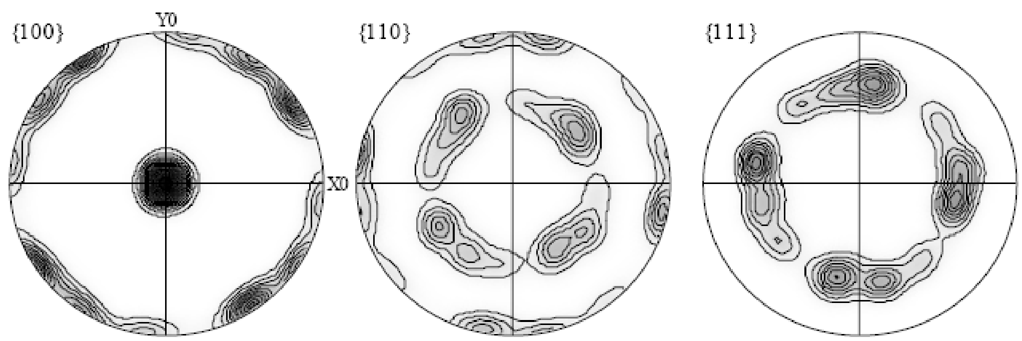

Fig. 9. Pole figures for machined research grade sample. Relative maximum intensity of 20.68.

Using the Orientation Distribution Function (ODF) the volume fraction (area fraction) of {100} grains parallel to the normal direction (zoning direction), misorientations up to 10 degrees allowed, for the as-zoned sample is shown in Figure 10. This value was calculated to be 61.3%. For the machined sample 98+% of the volume has {100} grains parallel to the normal direction within the 10 degree range. In Figure 10 only those grains which satisfy the above criteria are colored red. The majority of the grains which do not satisfy this criteria are located on the outer diameter and would be removed by machining as indicated by the dashed circle.

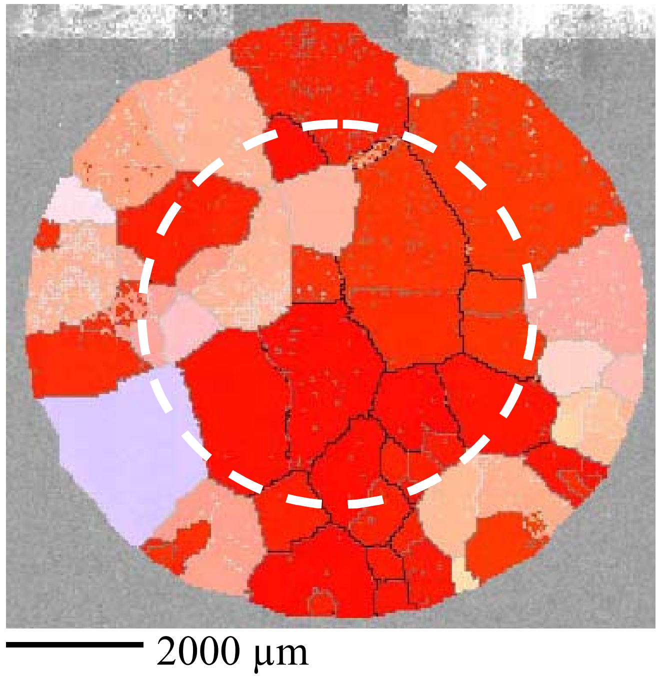

Fig. 10. As-zoned research grade sample with only those grains {100} oriented within 10 degrees filled in with red. Volume fraction calculated to be 61.3%.

Similar results are seen for the production grade samples. The main difference between the research grade results and the production grade results is that the degree of {100} orientation is not as strong in the production grade samples. Orientation maps of the production grade samples show more off-axis grains of various orientations than the research grade material.

The volume fraction (area fraction) of {100} grains parallel to the normal direction (zoning direction), misorientations up to 15 degrees allowed, for the as-zoned sample is shown in Figure 11. This value was calculated to be 29.3%. For the machined sample 44.1% of the volume (area) has {100} grains parallel to the normal direction within the 15 degree range, as shown in Figure 12. In Figure 11 and 12, only those grains which satisfy the above criteria are colored red. Once again, the majority of the grains which do not satisfy this criterion are located on the outer diameter and would be removed by machining as indicated by the dashed circle.

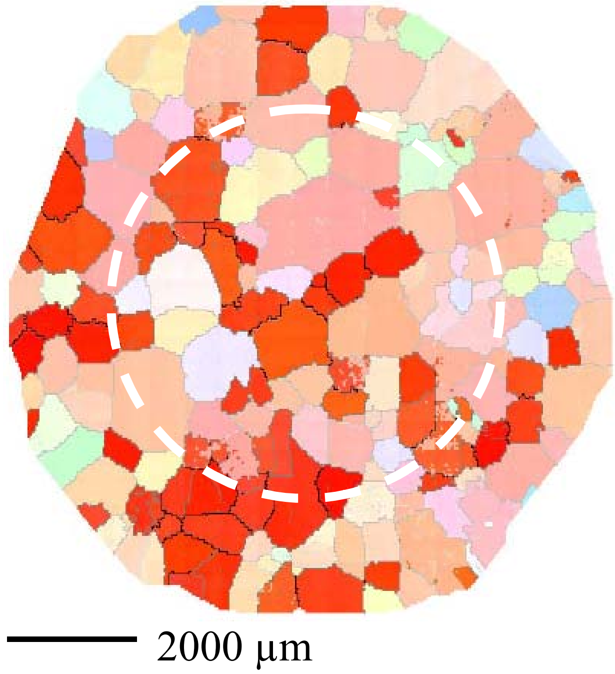

Fig. 11. As-zoned production grade sample with only those grains {100} oriented within 15 degrees filled in with red. Volume fraction calculated to be 29.3%.

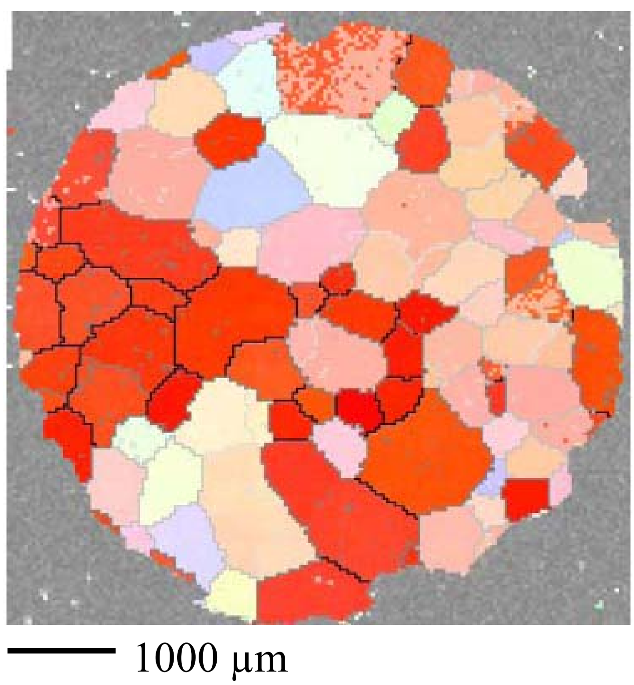

Fig. 12. Machined production grade sample with only those grains {100} oriented within 15 degrees filled in with red. Volume fraction calculated to be 44.1%.

3.2 Research and production grade Galfenol mechanical properties

Eight-research grade and twenty-production grade Galfenol samples were tested at room temperature, under static conditions, to determine their modulus, ultimate strength, and elongation. A 20,000 lbf load cell and a constant cross-arm velocity of 0.025 mm/minute (0.001 in./minute) were used throughout the testing on all samples. This testing rate is the same as was used by Kellogg[9] on single crystal Galfenol samples. This allows for a direct mechanical property comparison between polycrystalline and single crystal Galfenol.

The fracture mechanism for both research and production grade Galfenol appears to be intergranular and not transgranular. Similar observations by Kellogg[9] on a directionally solidified, polycrystalline, $Fe_{83}Ga_{17}$ sample supports this type of brittle fracture mechanism. During machining, the sample also fractured along grain boundaries, however, individual grains were found to exhibit a high amount of ductility as attempts to fracture them were unsuccessful. Other factors that could be affecting the ductility are the strain rate used and environmental factors. Past work has shown that the presence of hydrogen, from water vapor decomposition, can cause hydrogen embrittlement of the material. Both factors have been known to significantly affect the mechanical properties of Fe-aluminides[11, 12].

The eight research grade samples had modulus, ultimate strength, and elongation[13] averages of: 72.4 GPa (10.5 Msi), 370 MPa (53.7 ksi), and 1.2% respectively. Production grade Galfenol had the following mechanical property averages: modulus of 86.3 GPa (12.5 Msi), ultimate strength of 348 MPa (50.5 ksi), and an elongation of 0.81%. For reference, a [100] Galfenol single crystal[9] had a modulus value of 65 GPa (9.4 Msi), an ultimate strength of 515 MPa (74.6 ksi), and an elongation of 2%. As one would expect the research grade Galfenol, having a significantly higher degree of {100} orientation, would exhibit properties more in line with the single crystal results. Figure 13 shows a typical stress-strain curve for a research grade Galfenol sample.

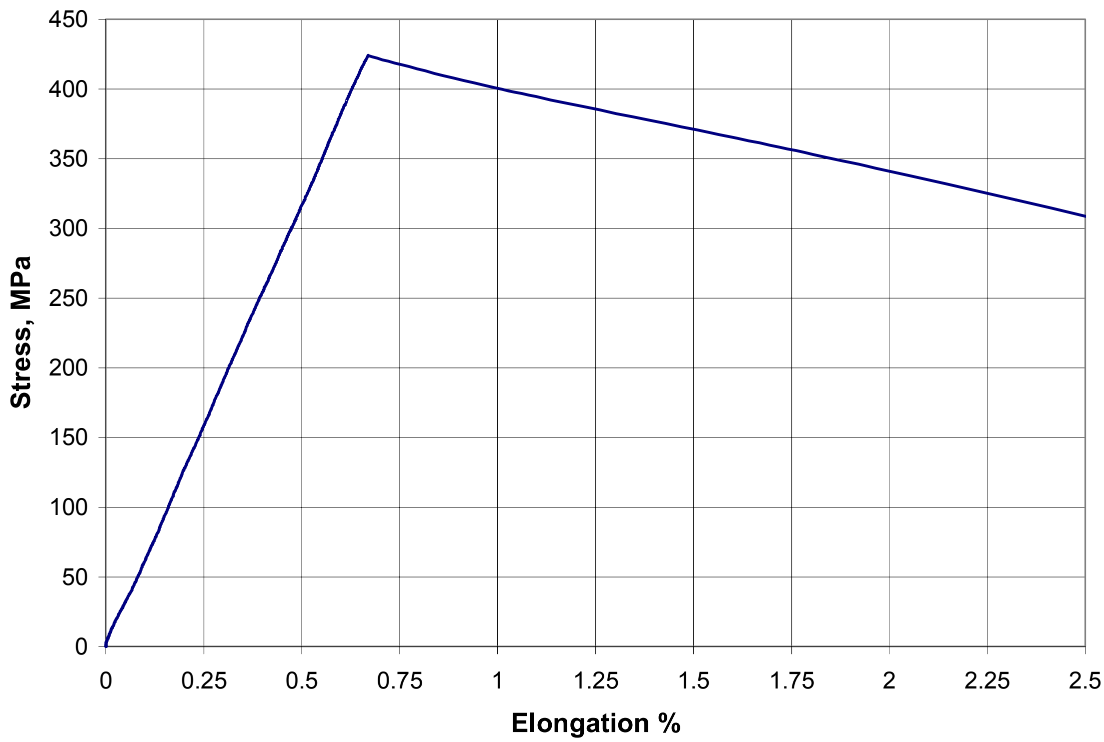

Fig. 13. Typical stress-strain curve for a research grade Galfenol sample.

3.3 FSZM vs. Bridgman growth magnetostriction

Figure 14 plots the magnetostriction versus applied field for research and production grade Galfenol produced using FSZM along with two samples produced using the Bridgman method[11]. The Bridgman samples were composed of 19 at% Ga, while the FSZM Galfenol was composed of 18.4 at% Ga. No post heat treatments were applied to any of the samples. The [100] single crystal specimen had a 3º misorientation with respect to the rod axis, while the multi-crystal sample was composed of large elongated grains with a 25º misorientation. The multi-crystal specimen is very similar in microstructure and orientation to the FSZM research grade Galfenol.

Figure 14 shows that the FSZM research grade Galfenol can have magnetostrictions greater than 250 ppm and approach the Bridgman single crystal value of 290 ppm. The FSZM production grade Galfenol does have significantly lower magnetostrictions at 175 ppm; however, it is produced at rates an order of magnitude greater than the research grade material or the Bridgman material thus providing a source of Galfenol at a significantly lower cost.

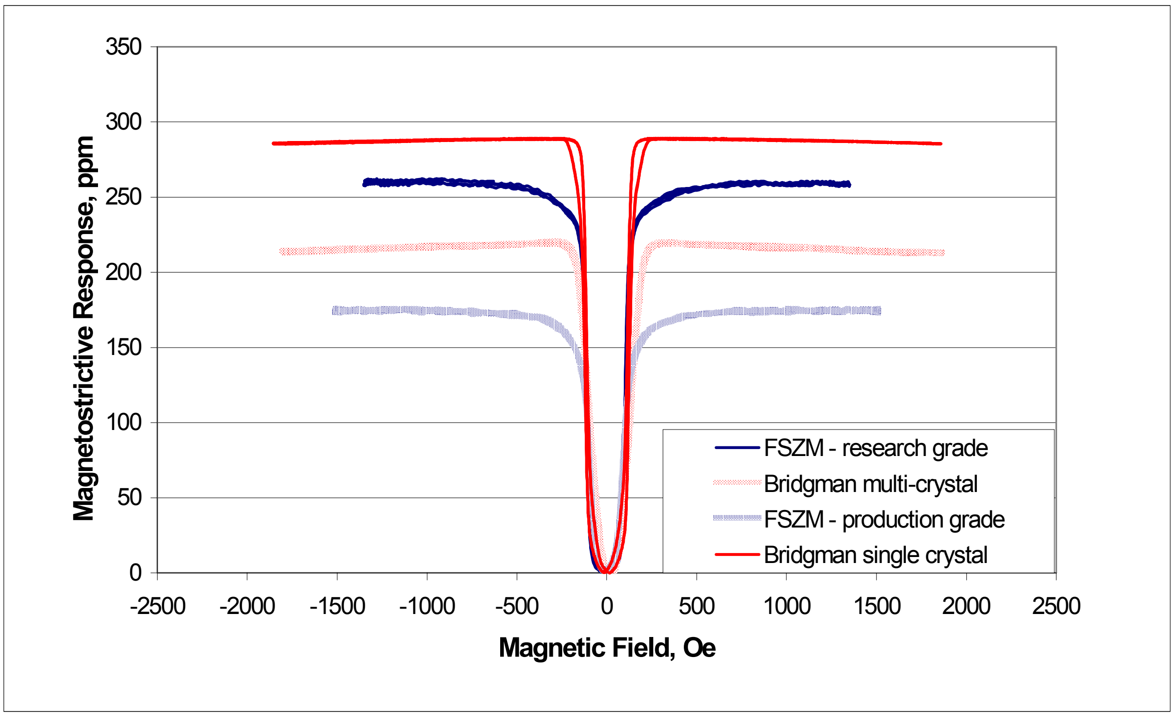

Fig. 14. Magnetostriction versus applied field for FSZM and Bridgman Galfenol samples.

4. CONCLUSIONS

FSZM has been used to produce Galfenol alloy, with a composition of $Fe_{81.6}Ga_{18.4}$, using zoning rates at least an order of a magnitude faster than single crystal Galfenol alloy produced using the Bridgman growth method. Research grade Galfenol is defined as Galfenol zoned at rates in excess of 25 mm/hr, while production grade Galfenol is defined as Galfenol zoned at rates in excess of 350 mm/hr. Conventional Bridgman growth rates are typically 2 mm/hr to 4 mm/hr.

The FSZM Galfenol exhibits a columnar microstructure with sub grain boundaries clearly evident in the production grade material. As expected the faster zoning rates resulted in a smaller grain size, 630 µm ± 85 µm for production grade material and 1350 µm ± 150 µm for research grade material. Powdered x-ray diffraction techniques showed that the ordered DO_3 phase, known to reduce magnetostriction, was not present in this material.

Machining the as-zoned Galfenol caused a significant increase in magnetostriction in both the research grade and production grade material. This increase was attributed to the removal of off-axis grains that had solidified in the outer diameter of the Galfenol rods. OIM was used to show how the removal of the outer area caused a large increase in the preferred {100} orientation and as a result the magnetostriction increased. For the research grade material a 15% average increase (220 ppm ± 25 ppm) in magnetostriction resulted from machining. For the production grade material an average increase of 15% (168 ppm ± 18 ppm) was seen after machining. The saturation magnetization of both types of materials was ~1.5 tesla. Single crystal Galfenol of a similar composition had a measured magnetostriction of ~290 ppm, with no post-growth heat treatments.

Static mechanical testing was completed on several research and production grade specimens. Both types of Galfenol exhibited brittle intergranular fracture which accounts for their poor ductility with respect to similar alloys. Investigations into the effect strain rate, hydrogen embrittlement, oxygen content at grain boundaries, and the presence of Ga-rich intermetallics at the grain boundaries has on the mechanical properties/fracture mechanics need to take place for further understanding. The research grade material exhibited mechanical properties (modulus of 72.4 GPa, ultimate strength of 370 MPa, and 1.2% elongation) more in line with [100] oriented single crystal than the production grade material (86.3 GPa, 348 MPa, 0.81% elongation).

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